

THE INSTITUTE SPOKESMAN



ANILINE POINT APPARATUS

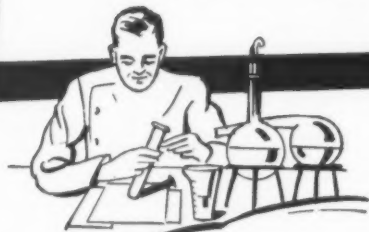
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VOLUME XI • NUMBER 12 • MARCH 1948



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"PRECISION" ANILINE POINT APPARATUS A. S. T. M. D611-47T.

A new, compact unit, for determining the aniline point of petroleum products and organic solvents according to A.S.T.M. D-611-47T. Both "Thin-Film" and "U-Tube" units are assembled in interchangeable bakelite cover. Changes from "U-Tube" to "Thin-Film" or reverse are easily and quickly made.

Application

Laboratory analysis and plant control of petroleum solvents such as Rubber solvent, V.M. & P., Naphtha, Stoddard solvent, and mixtures of aromatic solvents with petroleum naphthas.

Paint and varnish industries, to control proper blending of solvents to give not only solubility of various resins and gums, but also to control drying time of the finished product.

Large users of the new ester solvents and higher boiling alcohol solvents use aniline point to control solubility and compatibility of these solvents for synthetic resins, nitrocellulose, etc.

Method of operation:

Equal volumes of the sample and aniline are mixed, heated at a controlled rate, stirred until homogeneous, then cooled at a controlled rate until the cloud point is noted. The temperature at which the mixture becomes clear thru-out is the "aniline point". Procedure is covered by A.S.T.M. D-611-47T.

Features

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2. Bakelite cover assembly of both "Thin-Film" and "U-Tube" apparatus lifts from bath as complete units and can be changed over in seconds.
3. Compact induction motor, with belt drive, operates individual stainless steel stirrers for sample and bath liquid.
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6. Support rod $\frac{1}{2}$ " x 24" and two dovetail clamps included with equipment.

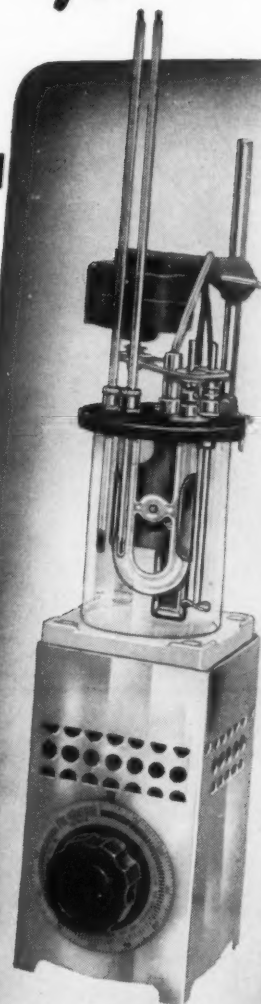
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ABOUT THE COVER

ANILINE POINT APPARATUS

The S.I.L. U-tube Aniline Point Apparatus is used to determine aniline points of petroleum products and other organic solvents. Recently adopted as a standard by the ASTM, in method D 611-47T, the U-tube apparatus may be used for clear, light-colored, or moderately dark samples.

The aniline point gives a rough estimation of the aromatic and paraffinic content of a solvent. The determination consists of mixing equal volumes of aniline and sample, heating at a controlled rate until homogeneous, and then cooling at a controlled rate until the cloud point is noted. The temperature at which the two phases separate is the "aniline point." This change is observed through a small window in the tube, with a light behind it.

The bath (usually oil) is stirred simultaneously with the sample—aniline mixture by means of a small motor with two pulleys. The rate of heating is regulated by the Ful-Kontrol heater, and cooling is regulated by the special cooling coil.

"Technical Committee Column"

This is the second issue of the Technical Committee Column. We hope that before the deadline of the third issue arrives we shall have received a "pile" of material from the membership of the Committee. We again call attention to the fact that the Technical Committee includes representatives of raw material suppliers, grease manufacturers, grease dispensing equipment manufacturers and grease consumers. This column, therefore, can be a convenient means for carrying on an open forum on the many problems facing the industry.

Progress has been made on the Committee's project entitled "Delivery Characteristics of Dispensing Equipment for Lubricating Greases." Organization of the Panel is under way and its present membership is the following:

Mr. L. C. Brunstrum, Standard Oil Company (Indiana).

Mr. R. P. Field, Balcrank, Inc.

Mr. Gus Kaufman, The Texas Company.

Mr. C. H. Mueller, Lincoln Engineering Co.

Mr. H. A. Murphy, Gray Co., Inc.

Mr. C. F. Raisch, Stewart-Warner Corporation.

The INSTITUTE SPOKESMAN

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GREASE INSTITUTE

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4638 Millcreek Parkway
Kansas City 2, Mo.

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Mr. T. G. Roehner, Socony-Vacuum Laboratories.

Mr. G. A. Williams, Battenfeld Grease & Oil Corporation.

The Panel is now being polled to determine the most convenient date and place to hold the first meeting. This, therefore, would be an opportune time for all members interested in this project to send in their suggestions and comments, so that the Panel may be properly guided in drawing up their program.

Relative to the activities of the ABEC-NLGI Cooperative Committee on Grease Test Methods, organization of a Subcommittee to investigate methods for the determination of dirt count of greases for anti-friction bearing greases has been completed. The membership of this Subcommittee is the following:

Mr. N. J. Gothard, Sinclair Refining Company (Chairman of Subcommittee).

Dr. E. W. Adams, Standard Oil Company (Indiana).

Mr. C. J. Boner, Battenfeld Grease & Oil Corporation.

Mr. J. E. Dillon, Aetna Ball & Roller Bearing Manufacturing Company.

Mr. O. L. Maag, Timken Roller Bearing Company.

Mr. E. E. Wagner, Hoover Ball & Bearing Company.

Interest in this project was prompted by experience of the laboratories of both ball bearing and grease manufacturers with the dirt count requirements included in Army-Navy Aeronautical Specifications AN-G-15a and AN-G-25 and also Bureau of Ordnance Specification 14G8 (Ord). The study of this subject actually has been sub-divided. ABEC has undertaken the assignment of defining the permissible limits of dirt count in greases furnished for a number of applications, particularly for prepacked bearings. The aforementioned Subcommittee will study methods for the determination of the number, size and nature of the foreign particles and will also submit recommendations concerning the interpretation of the data in terms of their practical significance. Any comments or suggestions from the members of the Technical Committee may be sent to either Mr. N. J. Gothard or Mr. T. G. Roehner and will be given careful consideration.

The Technical Committee has just received a new project, namely, Standardization of Metal Drums and Containers. This obviously will involve discussions with other associations, such as the American Standards Association. Members are invited to send in any information which will assist in guiding the Technical Committee's consideration of this problem.

The Motor-Driven Grease Worker and Its Application for Evaluating Consistency Stability of Lubricating Greases

by CARL W. GEORGI

Technical Director, Research Lab., Quaker State Oil Refining Corp., Buffalo, N. Y.

Presented at a Meeting of Technical Committee G on Lubricating Grease Held in Washington, D. C., January 17, 1947.

Report of Section II on Consistency Measurements and Related Physical Tests for Lubricating Greases of Technical Committee G on Lubricating Grease of Committee D-2 on Petroleum Products and Lubricants

Most lubricating greases become softer and acquire a lower consistency upon being subjected to working. This working may be considered as any handling or movement of the grease, such as filling of dispensers or guns, pumping through hoses, pipe lines, or fittings, as well as actual use in lubricating bearings or other mechanisms.

The scope of A.S.T.M. Tentative Method of Test for Cone Penetration of Lubricating Grease (D 217-47 T), contains several pertinent statements pertaining to working of greases and is quoted as follows:

"Scope:

"1. (a) This method describes three test procedures for the empirical estimation of the consistency of lubricating greases by measurement of the extent of penetration of a standard cone. The method is applicable to a variety of greases since it describes procedures for the measurement of worked penetration, of unworked penetration, and of block penetration. Penetrations up to 400, the practical limit of the cone, may be measured.

"(b) Unworked penetrations are affected by a number of factors which are difficult to control. They do not generally represent the consistency of greases in use as effectively as worked penetrations, which should be preferred wherever practicable, for evaluating lubricating greases.

"Note 1.—The unworked penetration of a grease may not be a reliable measure of its consistency in its original state of manufacture. Any transferring or other manipulating of grease subsequent to manufacture tends to affect the unworked penetration by amounts depending on the type of the grease and the handling it is given."

As mentioned, unworked penetration tests may be unreliable since different types of greases vary considerably in the

degree to which they undergo consistency change upon being worked. As a result, worked penetrations are preferred, and A.S.T.M. Method D 217-47 T calls for a 60-stroke working of the grease sample in the standard worker immediately prior to making cone penetration tests. It is generally recognized, however, that work-

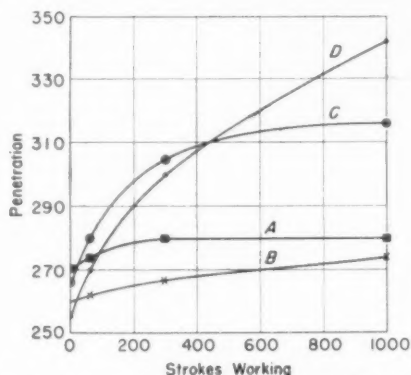


Fig. 1.—Penetration (Consistency) Change with Working in A.S.T.M. Grease Worker, Typical Types of Lubricating Greases.



Carl W. Georgi

ing of a grease sample for 60 strokes in the A.S.T.M. worker does not portray the complete story of the consistency stability of the grease. This is illustrated by Fig. 1 which shows consistency-working curves of four typical types of lubricating greases. Greases A and B are quite stable to working and display only moderate change or softening on working up to 1000 strokes in the A.S.T.M. worker. Grease C softened markedly up to 300 strokes working after which it became



Fig. 2.—Motor-Drive Unit with Workers of A.S.T.M. Dimensions Modified for Mechanical Rather than Hand Operation.

Courtesy of Precision Scientific Co.

relatively stable, whereas grease D displays progressive softening upon continued working with no indication of an "equilibrium point."

Because of the varied nature of lubricating greases in their reaction to extended working, many laboratories have adopted as more or less standard practice worked penetration tests after working up to 5,000 or 10,000 strokes and in some instances as high as 100,000 strokes. Figure 2 illustrates a motor drive unit together with workers of A.S.T.M. dimensions, but modified for mechanical rather than hand operation, as commonly used for such motorized working tests. Figure 3 is a drawing of another design of motor drive which accommodates two workers.

In view of the rather wide interest in motorized working of greases for intervals considerably longer than the 60 strokes specified in Method D 217, Technical Committee G on Lubricating Grease of Committee D-2 on Petroleum Products and Lubricants instituted a cooperative test program to explore the general subject. Three reference grease samples were distributed for cooperative testing:

Sample GII-2—No. 1 consistency, calcium-base grease, (U. S. Army Specification 2-107 Type).

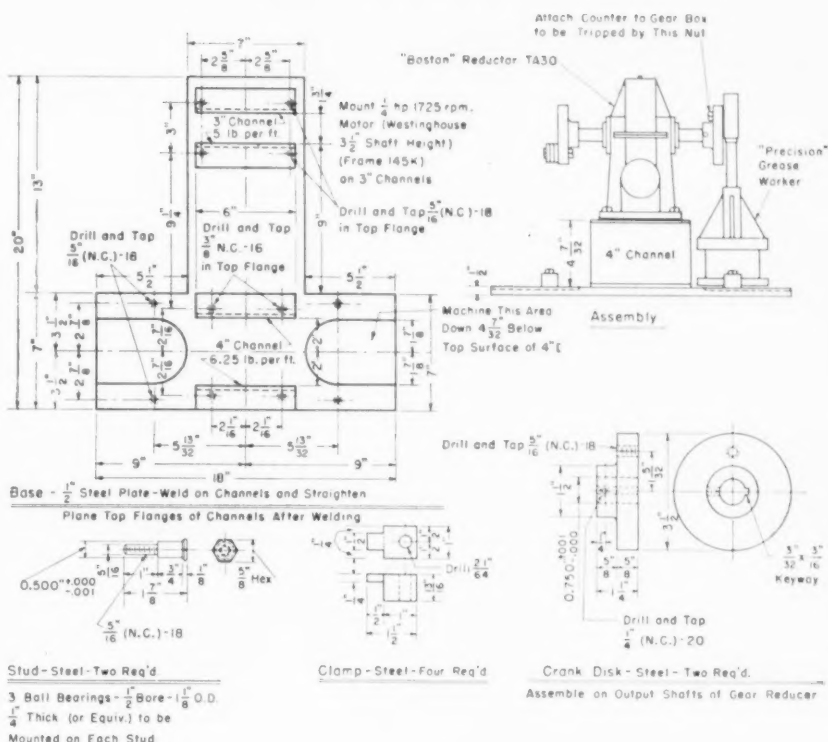


Fig. 3.—Schematic Drawing of Motor-Drive Design to Accommodate Two Workers.

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Sample GII-3—No. 2 consistency, soda-base grease, (U. S. Army Specification 2-108B Type).

Sample GII-4—No. 3 consistency, soda-base grease, high-viscosity mineral oil.

Fourteen laboratories submitted test results on motorized working of the three reference samples, with working up to 10,000 strokes. Several of the laboratories ran repeat tests on different dates so that a total of 19 sets of data on each grease sample were supplied. Table I summarizes the results. For comparison, Table II lists unworked and worked (60 strokes) penetration tests on the same greases by the standard A.S.T.M. Method D 217 (results from 18 cooperating laboratories).

The motorized worker tests show in general somewhat poorer inter-laboratory reproducibility than the regular penetra-

tion tests performed according to Method D 217, particularly with working above 300 strokes. This poorer degree of reproducibility is probably due to the higher temperatures developed in the grease samples during extended working, as shown in Table III. Temperature rise in the grease samples became quite significant in many of the tests reported, particularly with the longer working intervals. Two opposing factors are thus involved in motorized working. First, long working induces considerably higher grease temperatures than the 77 ± 3 F. specified for penetration testing in Method D 217. This may readily produce softer greases and correspondingly higher penetration readings. Second, the grease sample must be tested as soon as possible after working so that there will be no "set-back," as could be the case if a

warmed-up worked sample were cooled down to 77 F. prior to penetration testing. Evidently some of the cooperating laboratories made some provision for cooling the grease samples either during or after the motor working intervals, whereas others worked the greases and then made the penetration tests immediately thereafter with temperatures as developed. A detailed study of the cooperative test data indicates that inter-laboratory reproducibility with the motorized working penetration tests was as good as that attainable with the regular Method D 217 when the worked samples were maintained below about 87 F. (10 F. rise). When the samples warmed up above about 90 F., penetration readings tended to be higher than average and caused greater spread and poorer reproducibility of test results. This tends to be confirmed by several of the cooperating laboratories who supplied comparative test data on 60-stroke, worked consistencies using both hand and motor working, as summarized in Table IV. With only 60-stroke working, excessive temperature rise was not a factor, and the test results with either hand or motor working agree well within the limits of test reproducibility.

It would appear accordingly, with adequate provision for controlling grease temperatures in a properly limited range

TABLE I.—SUMMARY OF RESULTS OF COOPERATIVE PENETRATION TESTS—MOTORIZED WORKING OF REFERENCE GREASE SAMPLES.

	60 Strokes	300 Strokes	1000 Strokes	5,000 Strokes	10,000 Strokes
GREASE GII-2					
Average penetration, 19 tests.....	325	330	330	330	330
Maximum penetration	330	334	339	343	345
Minimum penetration	318	319	321	319	320
Spread	12	15	18	24	25
Percentage of Tests Within:					
± 5 Points of average.....	82	77	65	59	59
± 10 Points of average.....	100	94	100	77	88
± 15 Points of average.....	100	100	100	100	100
GREASE GII-3					
Average penetration, 19 tests.....	275	305	325	345	350
Maximum penetration	288	327	341	359	362
Minimum penetration	264	273	306	326	334
Spread	24	54	35	33	28
Percentage of Tests Within:					
± 5 Points of average.....	47	41	29	47	47
± 10 Points of average.....	77	65	70	70	82
± 15 Points of average.....	100	82	88	94	94
GREASE GII-4					
Average penetration, 19 tests.....	240	250	260	275	285
Maximum penetration	251	263	283	305	308
Minimum penetration	228	239	246	256	266
Spread	23	24	37	49	42
Percentage of Tests Within:					
± 5 Points of average.....	59	65	41	35	29
± 10 Points of average.....	82	82	70	65	77
± 15 Points of average.....	100	100	88	77	82

TABLE II.—SUMMARY OF RESULTS OF COOPERATIVE PENETRATION TESTS—UNWORKED AND WORKED PENETRATIONS BY A.S.T.M. METHOD D 217.

	GII-2	GII-3	GII-4
Summary of Unworked Penetration Tests			
Average penetration, 34 tests.....	320	225	220
Maximum penetration	328	236	234
Minimum penetration	304	210	208
Spread	24	26	26
Percentage of Tests Within:			
± 5 Points of average.....	65	68	47
± 10 Points of average.....	95	91	73
± 15 Points of average.....	100	100	100
Summary of Worked Penetration Tests (60 Strokes)			
Average penetration, 34 tests.....	325	280	240
Maximum penetration	333	296	255
Minimum penetration	311	266	230
Spread	22	30	25
Percentage of Tests Within:			
± 5 Points of average.....	68	62	56
± 10 Points of average.....	95	86	89
± 15 Points of average.....	100	97	100

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TABLE 1.—COOPERATIVE MOTORIZED WORKER TESTS—TEMPERATURE RISE AND TEST TEMPERATURES REPORTED BY COOPERATING LABORATORIES.

Temperature Rise, deg. Fahr.	No. of Tests				
	60 Strokes	300 Strokes	1000 Strokes	5000 Strokes	10,000 Strokes
Temperature Rise of Grease Sample During Working					
0 to 4.....	9	10	7	6	6
1 to 4.....	30	17	9	2	5
5 to 8.....	10	15	20	10	6
9 to 12.....	3	6	6	10	13
13 to 16.....	3	6	10	11
17 to 20.....	3	5	3
21 to 24.....	5	3
25 to 30.....	2	5
Grease Sample Temperatures When Penetration Tested After Working					
71 to 75.....	7
81 to 84.....	4	6	8	4	6
85 to 88.....	2	5	12	7
89 to 92.....	4	1	3	6
93 to 96.....	2	1	4	5
97 to 100.....	1	1	1	2
100 to 110.....	2	2
(All other tests made at 77 ± 3 F.)					

the motorized worker test can be as reproducible within and between laboratories as regular penetration tests made according to Method D 217. However, considerable additional cooperative test and development work would be necessary before this end could be achieved. In its present state of development, the procedure for motorized working of greases for longer than 60 strokes is not

sufficiently precise or reproducible to be included as a part of Method D 217. The results of the cooperative tests are accordingly being reported herein so that the information secured to date will be available to those interested.

It seems desirable to mention at this point that worked penetration tests on greases for intervals of several thousand strokes require careful and cautious in-

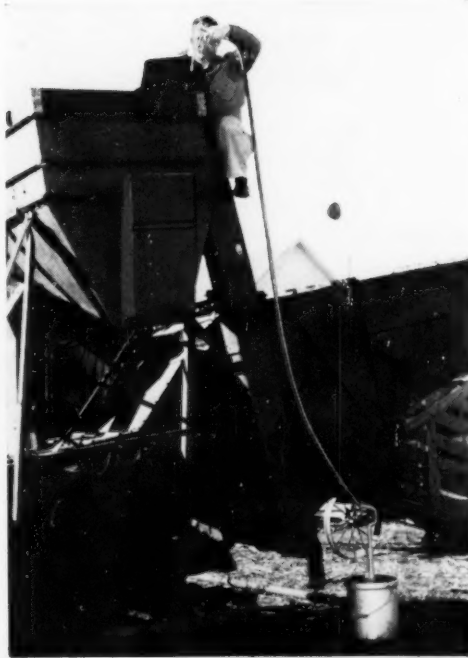
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TABLE IV. COMPARISON OF 60-STROKE WORKED CONSISTENCIES WITH HAND AND MOTOR-DRIVEN WORKERS.

Laboratory	Penetration Readings					
	GII-2		GII-3		GII-4	
	Hand Worked	Motor Worked	Hand Worked	Motor Worked	Hand Worked	Motor Worked
P-1.....	324 333	328 330	280 274	268 272	244 243	240 246
P-3.....	317 322	318 318	269 270	266 271	230 232	235 243
P-8.....	317 320	319	267 279	264	237 241	246
P-13.....	328	325 322	280	267 266	249	238 242
P-14.....	325	330	324	276	234	238
P-15.....	313 322	282 324	284 285	287 283	233 234	239 238

TABLE V. COMPARISON OF MOTOR WORKER TESTS WITH A.S.T.M. WORKER PLATE AND MODIFIED WORKER PLATE.

Strokes Working	Penetration Readings			
	Grease GII-2		Grease GII-4	
	A.S.T.M. Plate, 51 Holes, $\frac{1}{4}$ in.	Modified Plate, 325 Holes, $\frac{1}{16}$ in.	A.S.T.M. Plate, 51 Holes, $\frac{1}{4}$ in.	Modified Plate, 325 Holes, $\frac{1}{16}$ in.
60.....	326	328	241	243
300.....	330	330	257	252
1,000.....	332	330	265	278
5,000.....	335	334	280	316
10,000.....	335	334	285	330
100,000.....	337	346	310	

terpretation. Lubricating greases vary greatly in consistency stability or resistance to softening on working, as illustrated by Fig. 1, depending largely upon the kind and concentration of soap, the viscosity of the mineral oil constituent, and the details of processing during manufacture. Certain types of greases inherently have high resistance to softening on extended working, whereas other types inherently have quite limited resistance, but this factor alone, without considering many other important physical and performance properties, will not distinguish between a suitable or satisfactory lubricant for a given application and one which would be undesirable. While it might be considered at first glance that a grease displaying essentially no consistency change in motorized working tests might be the most desirable, this sort of conclusion may in many cases be quite incorrect. In many applications it is desirable that the grease soften and attain a state of near fluidity on the surfaces actually being lubricated. In fact, one of the reasons grease lubrication may be preferred over fluid oil may be that most greases retain their body or consistency only at points adjacent to and surrounding the lubricated moving parts and thus act as a seal to exclude dirt and moisture and to resist leakage. On the other hand, the grease feeding into and actually supplying the lubrication at the bearing surfaces is subjected to very high shear rates and may soften

to the point of supplying essentially fluid film lubrication with a minimum of viscous friction. As an additional factor, working even for many thousands of strokes in the A.S.T.M. worker might be the equivalent of only a few minutes of use in a bearing or similar close-fitted mechanism, since the rates of shear exerted in the A.S.T.M. worker are in the order of a few hundred reciprocal seconds at most, while the shear rates in an actual bearing may run into many thousand reciprocal seconds. Similarly, in service grease is normally subjected to considerable temperature variation, heating up during running and cooling off during idle periods. Such alternating cycles of heating and cooling may have a pronounced effect on the grease consistency and consistency change which is not measured or evaluated by motorized

worker tests wherein temperature fluctuation is in the order of only 50 to 80 F. and wherein temperature cycling is not encountered. In view of these considerations, many grease technologists have mentioned that motorized working tests have limited significance unless such have been carefully correlated with the particular application in question.

Modified Grease Worker With 1/16-In. Holes in Worker Plate

Certain grease specifications of the Federal Government agencies include a working test wherein the sample is motorized for 100,000 strokes in a modified A.S.T.M. grease worker, the modification consisting of a worker plate containing approximately 325 holes of 1/16 in. diameter instead of the customary 50 to 55 holes of 1/4-in. diameter.

One of the cooperating laboratories supplied comparative data on two of the reference grease samples, using the standard worker and the modified worker. Table V summarizes the test results. Grease GII-2, which possesses high resistance to breakdown on working, gave practically identical penetration test values with both worker plates. Grease GII-4, which has relatively poorer resistance to consistency change on work-

Continued on page 13

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Fig. 4.—New Grease Worker Incorporating Design Improvements.

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METALLIC SOAPS for GREASES

• By S. B. ELLIOTT Delivered before the 15th Annual N.L.G.I. Convention, Edgewater Beach Hotel, Chicago, Illinois, Oct. 16-18, 1947.

Continued from February Issue

Lead Soaps

Lead soaps are still used in substantial amounts for various applications where mild extreme pressure conditions are encountered. Though lead soaps in such circumstances are not functioning as thickeners but rather as a source of lead, they comprise an interesting class.

Though the lead salts of saturated and unsaturated aliphatic acids have been used, they have presented serious problems because of poor initial solubility or because of poor solubility after oxidation had occurred. As mentioned above, the lead salts of naphthenic acids, the carboxylic derivatives of cycloparaffin hydrocarbons, have become widely used as a result. The acids are saturated and stable to aerial oxidation and their lead salts, because of the high solubility of the cyclic structure, are soluble in a broad range of hydrocarbons.

Since the salts are primarily interesting because of the lead they contain, a mixture of lead di-naphthenate and lead mono-naphthenate is usually manufactured. The viscosity of such a concentrate having a given lead content is much lower and thus more easily handled whereas the solubility in hydrocarbons is quite adequate if the lead mono-naphthenate concentration is not too high.

As would be expected, the molar concentration of the mono-naphthenate can be higher when high-molecular-weight rather than low-molecular-weight naphthenic acids are used because of the large amount of nonpolar residue. If it rises too high, however, both the dispersion time and the permanent solubility in oil is affected.

Barium Soaps

It is not too long since barium soaps had little or no place as thickeners of hydrocarbons because barium compounds were higher in cost than those of calcium and offered little or no advantages. Thus, as much water was required for stabilization so that high-temperature operation was possible with neither barium nor calcium soap greases.

However, additional research⁴ demonstrated that mixtures of di-acid and mono-acid soaps are unique in that substantial quantities of water are not necessary for their stabilization. These soaps contained 40-60 per cent more barium than is required to form the normal, di-acid soap and as little as 0.1 per cent water is satisfactory though it may rise as high as 0.3-0.4 per cent. However, the presence of some glycerin as a peptizing agent is desirable so some glycerides should be used.

The greases prepared using these materials are unctuous, nonfibrous materials having relatively high soap contents which have given excellent performance under severe conditions, so that

4. Ott, T. F., Clarke, P. S., and Van Marter, C. H., U.S.P. 2,033,148 and 2,154,383.



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it appears definite that high soap concentrations can be satisfactory. Further, because of the small amount of water present, there is little change in structure on heating to 225° F. or higher and then cooling.

Through the use of the hydroxy soaps, it is possible to use a wide range of soap stocks and to use oils of varying types and viscosities without too much trouble.

Calcium Soaps

Calcium soaps are of great interest because of the tremendous volume used in the thickening of oils. They are simple to prepare using any of the usual methods of reaction, hydrated lime being alkaline enough to insure an almost complete reaction at high temperatures even when the soap is substantially diluted with oil. However, the temperature must be high enough to secure adequate reaction, since too much free fatty acid or free hydrated lime will lead to instability.

Since calcium stearate is less soluble in hydrocarbons than calcium oleate, the

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President's Column...

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They purchase a lubricant and expect it to perform a miracle. And I mean that in the literal sense of the word. Because only by means of a miracle could lubricants be expected to function properly when they are filled with dirt and grit



J. R. Corbett
President N.L.G.I.

and all matter of foreign substances. Yet the consumer, failing to care for his lubricants, applies them readily no matter what their condition. And when the grease fails to do its required duty, he's up in arms at—that's right, the grease manufacturer.

All our efforts to produce flawless and effective lubricating agents are useless if the consumer is not instructed in the care of the merchandise he buys.

Let's consider, for a moment, the tremendous amount of work that we put into the manufacturing of our products.

In the N.L.G.I., we have the skilled services of the Technical Committee—men who exert great energy in trying to perfect even more effective lubricants and ways of applying these lubricants. Thus we have the finest technical brains in the country working for us day after day, trying to improve products, methods and practices.

In our plants and factories we pride ourselves—and justly so—on our scientific manufacture and controlled uniformity of the products we sell. Our laboratories are veritable beehives of activity, humming night and day with research specialists, who are dedicated to

the cause of giving the consumer even better lubricants.

Our packaging methods are modern and up to the minute. The lubricants are packaged fast and, what's more important, they're packaged clean. We exercise the greatest of care in making sure the product goes into the package free from the slightest particle of dust, grit, dirt, or any foreign substance.

Great pains are then taken by the manufacturer and distributor in storing the finished products. Lubricants are packed away in a clean, dry place where they'll be unexposed to any extreme weather temperatures.

In other words, when the consumer buys our products, he gets lubricants that are completely adequate to do the job they were intended to do. But the reason the lubricant can fail to perform properly is because the consumer often fails to care for his products properly.

As grease manufacturers, we should keep this precept in mind at all times. If we want our products to sell, we've got to do more than sell the dealer. We've got to sell the consumer as well. And if

Continued on page 22



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THE MOTOR-DRIVEN GREASE WORKER

Continued from page 8

displays a considerable difference in generation change on working in the two types of workers. It is interesting that working of GII-4 for 100,000 strokes with the standard worker having holes of $\frac{1}{4}$ -in. diameter produced somewhat less softening of the grease than only 10,000 strokes working with the modified worker having 325 holes of $\frac{1}{16}$ -in. diameter.

Although the amount of test data comparing the standard worker with the modified unit is limited, it appears with greases having good resistance to breakdown on working, the number and size of holes in the worker plate make no material difference. With greases which are quite susceptible to consistency change on working it appears, however, that the number and size of holes in the worker plate have a significant effect in the results secured.

Improvements in A.S.T.M. Grease Worker

A by-product of the cooperative test program on motor working was the accumulation of experience which indicated a need for improvements in the design of the A.S.T.M. grease worker as

originally specified in Method D 217—44 T.

1. A pet-cock or surge tube should be incorporated on the worker cover to relieve pressure and to minimize inclusion of air in the grease sample during working.

2. The worker cover should be recessed to accommodate the worker plate and to leave the cup full of grease after the working period.

3. A single, large diameter plunger shaft and a heavier perforated plate should be incorporated to overcome weakness of construction as noticed particularly when working harder greases.

4. A packing gland should be added to the worker cover around the plunger to compensate for wear and to minimize inclusion of air or grease leakage.

5. Heavier threads or a bolted closure are desired to minimize crossing or stripping the threads, as sometimes encountered with the original worker.

6. The worker cup should be of one piece cast construction or welded to avoid breakage at the juncture of the base and the walls.

All of these design improvements have been incorporated in the new worker as specified in Method D 217-47 T, and as shown in Fig. 4. The new worker is also designed so that it may be operated by hand or by a motor drive unit.

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Sinclair Refining Co.	N. J. Gothard
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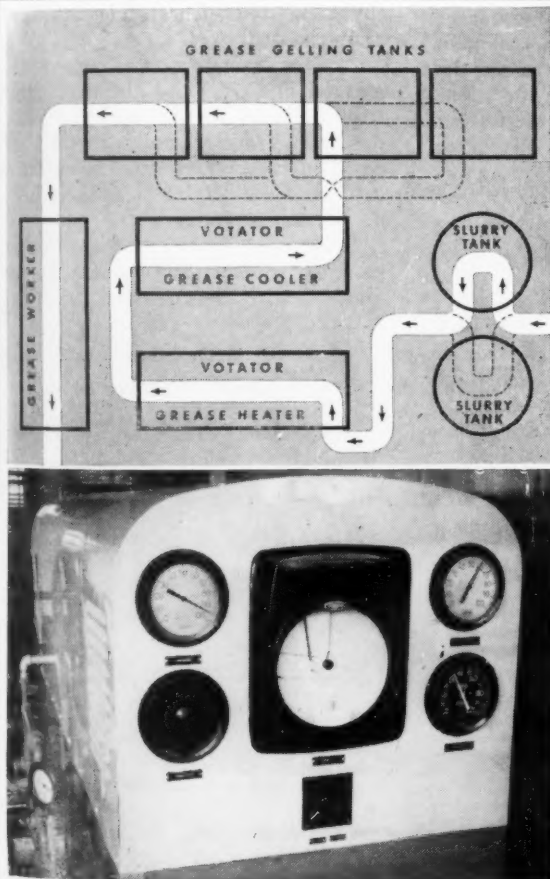
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PENETRATION of BLOCK GREASES

BY L. C. BRUNSTRUM AND A. W. WEITKAMP

Group Leader and Senior Project Chemist, Respectively, Standard Oil Co. (Indiana), Whiting, Ind.

Tentative Method of Test for Consistency of Lubricating Greases and Petrolatum (D217-44 T), 1946 Book of A.S.T.M. Standards, Part III-A, p. 846.

Data Presented at a Meeting of Technical Committee G on Lubricating Grease Held in Washington, D. C., January 17, 1947.

SYNOPSIS

A study of A.S.T.M. penetration values obtained on hard greases revealed that the results were critically dependent on the dimensions of the cone tip. The significant measure was found to be the cross-sectional area of the cone tip at the surface of the penetrated grease cake. The depth of penetration is a convenient though indirect measure of the cross-section. It follows that close dimensional tolerances must be employed in the manufacture of the tips if reproducible results are to be obtained.

In a recently completed cooperative program aimed at the improvement of the cone penetration method, thirteen laboratories tested five block greases under conditions as nearly identical as possible. Each laboratory used the approved A.S.T.M. grease penetrometer specified in A.S.T.M. Method D 217. The following laboratories cooperated: Quaker State Oil Refining Corp., Sinclair Refining Co., Standard Oil Co. (Ohio), Socony-Vacuum Oil Co., The Texas Company, Cities Service Oil Co., California Research Corp., Rock Island Arsenal, Union Oil Co. of California, Imperial Oil Co., Bendix Aviation Corp., Gulf Research and Development Co., and Standard Oil Co. (Indiana).

In Table I are listed the penetration values reported by the thirteen laboratories using specimens from a single block of each of the five block greases. Penetration numbers are arranged at the head of each tabulation and a code number corresponding to each laboratory is entered in the table under the appropriate penetration number for each grease. Each result is the rounded average of nine readings, three being taken on each of three adjacent sides of a carefully prepared cubical specimen. It will be noted that most of the results fall within 3 or 4 point ranges and that the laboratories reporting higher or lower results were quite consistently high or low. Laboratories Nos. 5, 12, and 13 each submitted two sets of results. The duplicate data from laboratory No. 13 were obtained by one operator using a single instrument but with one new and one old cone tip. These tips will be referred to below as tips *N* and *O*, respectively.

Detailed results listed in Table II show the precision obtainable with tips *N* and *O* on any given face of each specimen. Slight differences to be noted between different faces of a given specimen serve only to emphasize the need for averaging

values over three adjacent faces. It seemed significant that tip *O* consistently gave lower penetration, by about two penetration units, than tip *N*. Microscopic examination of the tips revealed that tip *O* had been chipped and had an abnormally sharp point, whereas tip *N* was a new tip well within the dimensional tolerances of Method D 217.

Photomicrographs of the tips are shown in Fig. 1. Tip *N* is at the left, and tip *O* in the middle. The images of tips *N* and *O* are shown superimposed at the right. The difference in length of the two tips is approximately 0.2 mm. or two penetration units. This striking correlation with the penetrations actually observed seemed to indicate that the sharpness or contour of the point has little effect on the penetration except in so far as it affects the setting of the penetrometer at the start of the test. Since the angles of tips *N* and *O* were identical it follows that these tips came to rest in any given specimen at equal projected cross-sectional areas. The results with tip *O* were less than those with tip *N* because any given cross-section is about 0.2 mm. nearer the point.

In order to test the effect of excessive truncation, tip *O* was repeatedly ground down and tested against a single face of a given sample of grease. Seven such grindings were made and each time the tip was found to come to rest in the

test specimen at the same cross-sectional area. In Fig. 2 is a series of photomicrographs, beginning at the left with a perfect new tip and showing in sequence the seven successive grindings of tip *O*. The base line common to all of the tips shows the depth of penetration into the test specimen. After the seventh grinding no additional grindings were performed which served to sharpen the tip but not to shorten it. Photomicrographs are shown in Fig. 3. Subsequently, additional flat grindings were made until a tip area was reached at which the cone simply rested on the sample surface without appreciably penetrating it. Photomicrographs of the last grindings were not made because the field of view of the microscope was too small. However, in Fig. 4 is a sketch showing schematically all of the tip shapes. *T* is the dimension from the theoretical apex to the flat end of the cone after each grind; *P* represents the average of three penetrations on a single small sample; *C* is equal to the sum of *T* and *P* and is the distance from apex to the cross-section at which the cone stops penetrating. The data used in constructing this diagram are shown in Table III. The constancy of the value of *C* for the twelve cone lengths, including two with sharpened points, is taken as proof that:

1. For a given sample of block grease any cone stops penetrating at a certain cross-sectional area.
2. The penetration as reported under Method D 217 is the distance from the truncated end to this area.
3. The sharpness of the tip is of no importance except to the extent that it affects the original setting of the tip on the sample surface.

Laboratory No. 7 is one that consistently reported low values. By subsequent actual measurement the diameter

P = average of three penetrations on a single small sample.

T = dimension from theoretical apex to the flat end of the cone after each grind.

C = sum of *P* and *T*, and its distance from apex to cross-section at which cone stops penetrating.

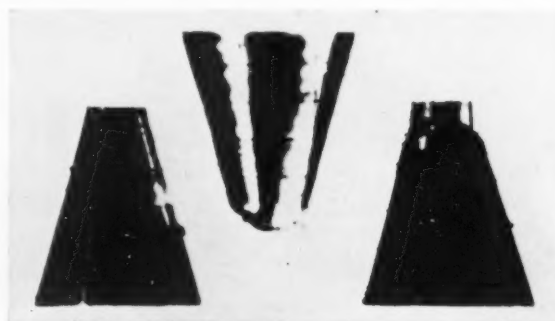


Fig. 1.—Photomicrographs of the Tips of the Cones.

TABLE I.—COOPERATIVE PENETRATION DATA.
G-II-J

Penetration	14	15	16	17	18	19	20	21	22	23	24	25	26	27
Laboratory (Original)	7						5	3 11	6	1 13A 8	6 12	10 12A	13 12A	2 4 9
(Corrected)												7 2		
G-II-K														
Penetration	9	10	11	12	13	14		15	16	17	18		19	20
Laboratory (Original)	7							3 6 8	5A 9 13A	1 4 12A	10 12 13		2	
(Corrected)											2		7	
G-II-L														
Penetration	23	25	26	27	28	29	30	31	32	33	34	35	36	37
Laboratory (Original)	7						3 11 8	1	6		5 5A 9 10 12 12A 13A	2 4 13		
(Corrected)											7 2			
G-II-M														
Penetration	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Laboratory (Original)	7			11				1 3 8			5 5A 13A	9 10 12A 13	4 12	2
(Corrected)											7	2		
G-II-N														
Penetration	12	13	14	15	16	17	18	19	20	21	22	23		
Laboratory (Original)	7			11	6	3	1 8	5 5A 12 13A	4 9 12A	2 10 13				
(Corrected)								2			7			

of the truncated tip used by laboratory No. 7 was 0.035 in. and the cone angle was 27 deg. This is equivalent to an excessive truncation of about 10 penetration units for the products tested. If this correction is added to the penetrations

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Fig. 2.—Photomicrographs Showing Perfect New Tip (Left) and Seven Successive Grinds of Tip O.



Fig. 3.—Photomicrographs Showing (Left) Seventh Grind of Tip O, and Two Additional Grinds Made to Sharpen, but Not Shorten the Tip.

TABLE II.—ACTUAL PENETRATION DATA, LABORATORY NO. 13

Sample	Tip N			Tip O			
	Top	Side	End	Top	Side	End	Difference
G-II-J.....	25	25	25	28	23	22	
	26	25	26	28	22	22	
	25	25	25	24	22	22	
Average.....		25.2			22.6		2.6
G-II-K.....	18	18	10	16	16	17	
	19	17	19	15	16	16	
	18	17	18	15	17	16	
Average.....		18.1			16.0		2.1
G-II-L.....	35	35	35	34	34	34	
	34	36	35	34	34	34	
	35	36	35	35	34	34	
Average.....		35.1			34.1		1.0
G-II-M.....	20	30	29	28	28	28	
	30	30	29	28	28	28	
	29	30	30	28	28	28	
Average.....		29.6			28.0		1.6
G-II-N.....	21	20	21	19	19	21	
	21	21	21	20	19	21	
	21	20	20	20	20	21	
Average.....		20.7			20.0		0.7

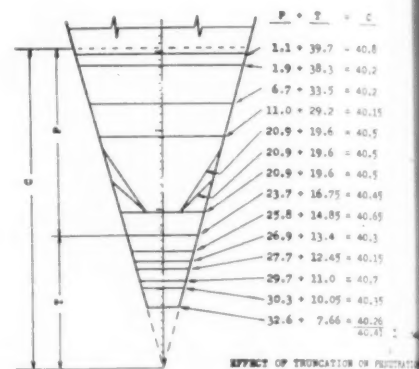


Fig. 4.—Schematic Diagram of All the Tip Shapes.



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originally reported by laboratory No. 7. The penetrations of this laboratory fall in line with the majority as shown by the corrected values in Table I.

On the other hand, laboratory No. 2 reported high values and we might assume that its tip had not been truncated to the extent required by Method D 217. Examination under the microscope disclosed that it had been damaged and re-ground to a sharper angle, the new angle extending well up along the tip. Therefore, no direct correction can be made. However, it is possible to calculate the area of the cross-section of the cone at

the point of penetration. This area would occur about 0.2 mm. too far up on the tip compared to a standard tip. The results of laboratory No. 2 are also in line if corrected by this amount. It is likely that the results from the remaining laboratories could be similarly corrected.

It was concluded that Method D 217 will be entirely satisfactory for block

greases if the cone tips are periodically measured for conformity, and lower tolerances on the tip are required in the revised method.

The tip manufacturer was consulted as to his ability to supply cone tips of lower tolerance and the following specifications were approved by Technical Committee G:

	Old Specification	New Specification
Length (after truncation), in.....	0.59 \pm 0.01	0.59 \pm 0.01
Base, in.....	0.33 \pm 0.005	0.33 \pm 0.002
Angle of cone, deg.....	30	30 \pm 1
Diameter of truncated tip, in.....	0.015 \pm 0.003	0.015 \pm 0.001
Material	Stainless or hardened steel	Hardened steel

TABLE III.—EFFECT OF TRUNCATION ON PENETRATION.

Test No.....	D-217 Tip	1	2	3	4	5	6	7	7a	7b	8	9	10	11	12
Tip diameter, in....	0.016	0.021	0.023	0.026	0.028	0.031	0.035	0.041	0.016a	0.016a	0.061	0.070	0.080	0.083	0.086
Penetration 1	32.0	30.0	30.0	27.5	26.9	26.0	24.0	21.5	21.2	20.9	11.4	6.5	1.7	1.0	1.0
	33.0	31.0	29.0	27.5	26.3	26.0	23.0	20.0	20.5	20.8	10.5	6.9	2.0	1.4	1.1
	33.0	30.0	29.0	28.0	27.3	25.3	24.0	21.1	20.6	21.5	10.5	6.7	2.0	1.0	1.0
	21.0	20.5	11.4
	20.5
Average	32.7	30.3	29.7	27.7	26.9	25.8	23.7	20.9	20.9	20.9	11.0	6.7	1.9	1.1	1.0
Truncation, mm./10	7.7	10.1	11.0	12.5	13.4	14.9	16.8	19.6	19.6	19.6	29.2	33.5	38.3	39.7	40.9
Penetration plus truncation	40.4	40.4	40.7	40.2	40.3	40.3	40.5	40.5	40.5	40.5	40.2	40.2	40.2	40.8	41.9

a Repaired from 0.041 to 0.016. Truncation calculated using 0.041 (see Fig. 21).

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METALLIC SOAPS FOR GREASES

Continued from page 10

oleate concentration may be raised so as to minimize bleeding of oil, an iodine value of 40 to 60 for the soap stock generally representing a high enough concentration. Calcium naphthenate, because of the cyclic nature of the naphthenic acids, is still more soluble in hydrocarbons than the oleate so that it is not much used as a thickening agent.

By very careful choice of the fatty acids used to prepare the calcium soaps, it is possible that as little as 1.0 per cent water may be necessary to stabilize the system. However, water or other polar compounds are always necessary, for nonpeptized soaps such as calcium stearate or oleate exhibit insolubility.

As much as 5.0 per cent water is some-

times used to stabilize the soap-hydrocarbon system but it appears likely that a substantial portion of this remains as discrete droplets through the mass, from 0.5 to 3.0 actually dissolving. As is well known, however, this water is most active when a controlled amount of excess fatty acid is present in the grease. If too much acid is present, the consistency of the grease is prone to be soft whereas too little leads to inadequate soap dispersibility.

It is interesting to note that it has been theorized⁶ that fibrillar crystallites of hydrated calcium oleate comprise the thickening agent in such greases, little or no interaction of soap and hydrocarbon occurring, this theory being based on an observation that the viscosity of such greases decreases very rapidly at the melting point of the soap. The water, designated as water of hydration, though the mode of bonding is uncertain, appears to function as a bridging agent between

crystals, assisting in the development of a definite yield value.

With the development of a continuous process for the manufacture of calcium greases,⁷ the importance of the amount of water present and dissolved has been clearly shown. In the continuous process, by closely controlling the water at about 12 per cent, based on the weight of soap, a lower percentage of soap was required to secure a given penetration. Further, because of fine dispersion of the water, excess was not required over that needed for peptization so that a clearer product resulted. It would be expected, the temperature at which the hydrated calcium soap concentrate was blended with the main oil must have had to be controlled carefully in order to secure optimum thickening.

As a total or partial replacement of the water, so as to secure stability at high temperatures, polar compounds such

5. Rhodes, F. H., and Wannamaker, T. E., Ind. Eng. Chem., 29 (6), 702 (1937).

6. Hoppler, Fette und Seifen, 49, 700 (1942).

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long-chain alcohols have had some effects. On the other hand, stability toward working involves not only chemical considerations but physical as well.

Sett greases are interesting examples of highly peptized soaps in hydrocarbon systems. Rosin, being relatively high in crystalline acids and low in high solvency terpene derivatives is unsatisfactory for the preparation of calcium soaps having high thickening powers. During the preparation of rosin oil, however, it appears that cracking of the rosin acids molecules occurs, accompanied by the formation of rather active terpene-derivative peptizers. Peptization thus substantially increases the solubility of the rosin and soaps and makes possible the development of thickened systems.

As still another type of peptization is the interaction of various soaps with each other so as to change substantially the thickening power of the mixture as compared with either of the soaps. Thus, sodium soaps peptize aluminum

soaps, calcium soaps appear to peptize lead soaps, etc. It is not definite how the soaps interact but some observations indicate that bonding may occur with the formation of the equivalent of double salts. One effect of this interaction is to necessitate close observation of the purity of the various soaps, but properly applied the same effect can be used to modify the hydrocarbon-soap structure in desirable ways.

Lithium Soaps

Lithium soaps are unusual because of their marked divergence in properties from those usually expected of the alkali metal soaps. Probably this difference in behavior occurs because of the small size of the lithium atom as compared to the other alkali metals. Thus, properly formulated lithium soap greases are satisfactory for use between $+400^{\circ}$ F. and -90° F., a range which is unequaled by other materials. As is well known, their water resistance equals that of the greases from soaps of polyvalent metals because

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of the water insolubility of lithium soaps. Their high melting point reflects the high melting point of the lithium salts as compared to the relatively low melting points of the soaps of polyvalent metals.

Usually high-grade double or triple-pressed stearic acid is used, though this almost always contains quantities of palmitic acid. However, reasonable amounts may be acceptable since extremely poor gelation does not begin to appear at that chain length. However, oxida-

tion products, unsaturated acids, glycerides and acids of substantially different chain length than stearic or palmitic may modify the penetration of the finished grease to an undesirable degree. Thus, the soaps of a homologous series of saturated aliphatic acids showed that the stearate produced greases exhibiting the least bleeding, the most consistency stability, and the best consistency-soap concentration relationship when naphthenic stock was used.⁸ However, when

a paraffinic stock was used, the palmate performed best.

The acids react readily with lithium hydroxide but it is still important that the reaction be taken virtually to completion. Because of the high melting point of lithium stearate of approximately 216° C., it is rather important that crystallinity of the product be kept at a minimum so that oil dispersability

8. Luckenbach, Jr., W. F. and Meech, Jr., H. Inst. Spokesman, 10 (4), 2 (1946).

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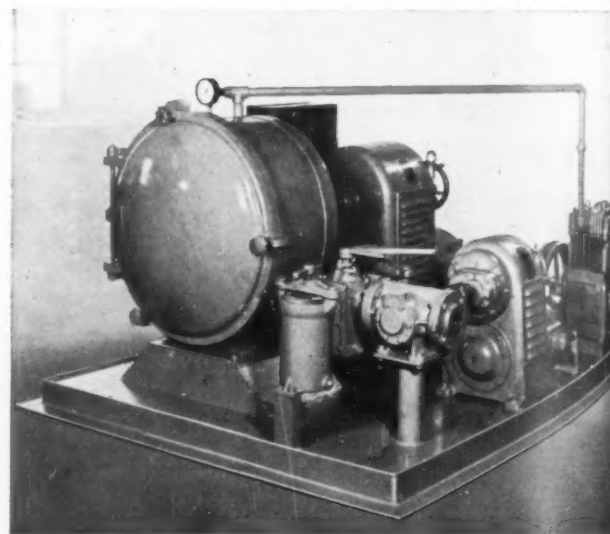
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This degree of crystallinity is, of course, indicated to some degree by the melting value.

So as to obtain reproducible thickening it is essential to keep the water concentration in the soap below 1.0 per cent and metals which function as oxidizing catalysts must be kept at trace concentrations in order to secure good oxidizing stability. Further, cations and anions which are not deleterious because of their oxidizing activity can contribute undesirable instability to lithium greases so far as consistency and melting is concerned.

So as to avoid an uncontrolled amount of gelatin, a limited quantity of free fatty acid is required, usually 0.1 per cent minimum and 0.25 per cent maximum (as oleic) proving satisfactory. A temperature of approximately 390° F. is usually adequate to disperse the soap in the oil, rapid cooling of the mixture producing an acceptable product.

Sodium Soaps

Sodium soaps, because sodium is a monovalent metal, are much simpler to prepare than those of polyvalent metals but the physical chemistry of their hydrocarbon solutions is quite as complicated. Simple neutralization of the usual monobasic acids produces either the normal salt or acid salt depending on the amount of sodium hydroxide used.

Dehydration to the anhydrous soap then produces a base material for thickening hydrocarbons.

Heating the anhydrous sodium soap to a high temperature with the oil, however, does not produce a desirable grease structure in the absence of glycerol. However, when a small percentage of water is present in the same mass, solution of the soap at high temperature is noted with the formation of an acceptable gel on cooling.

It would appear the peptization is the most important factor in this activity but the formation of hydrates causing

a substantial change in the phase diagram also probably occurs. Work with sodium oleate⁹ has demonstrated that peptization alone was adequate to cause development of an acceptable structure with the soap of such an unsaturated acid.

⁹ Rhodes, F. H., and Allen, H. D., Ind. Eng. Chem., 25 (11), 1274 (1933)

¹⁰ Puddington, I. E., Inst. Spokesman, 9 (9), 1

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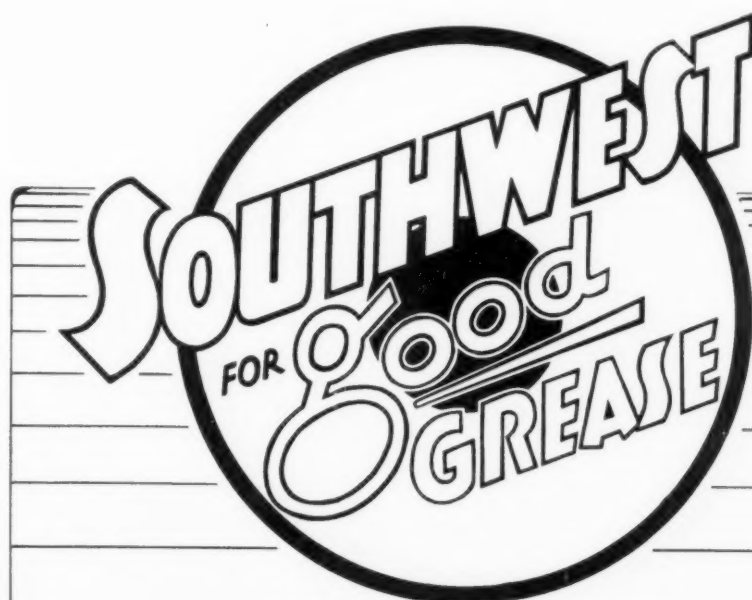
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